



Novel developments and future needs in methods of analysis for trace element species in food and feed control

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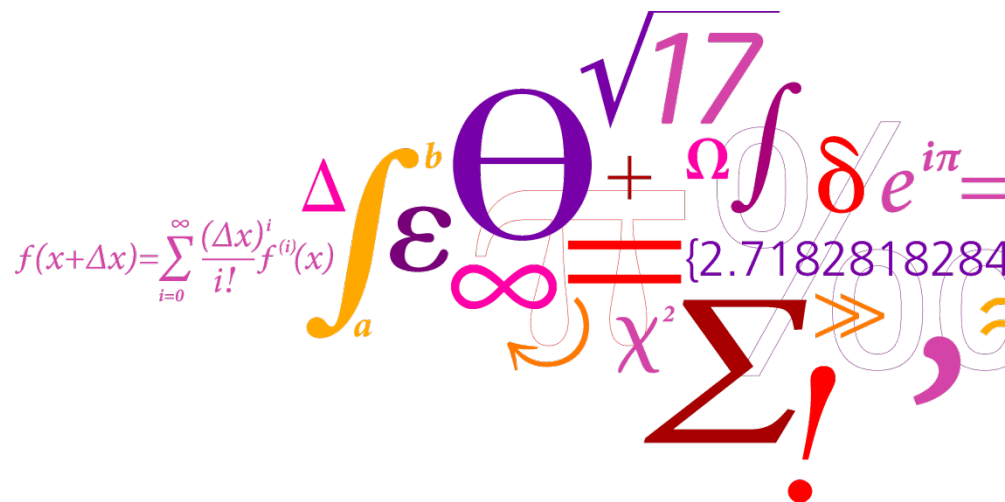
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Novel developments and future trends in methods of analysis for trace element species in food and feed control

Jens J. Sloth

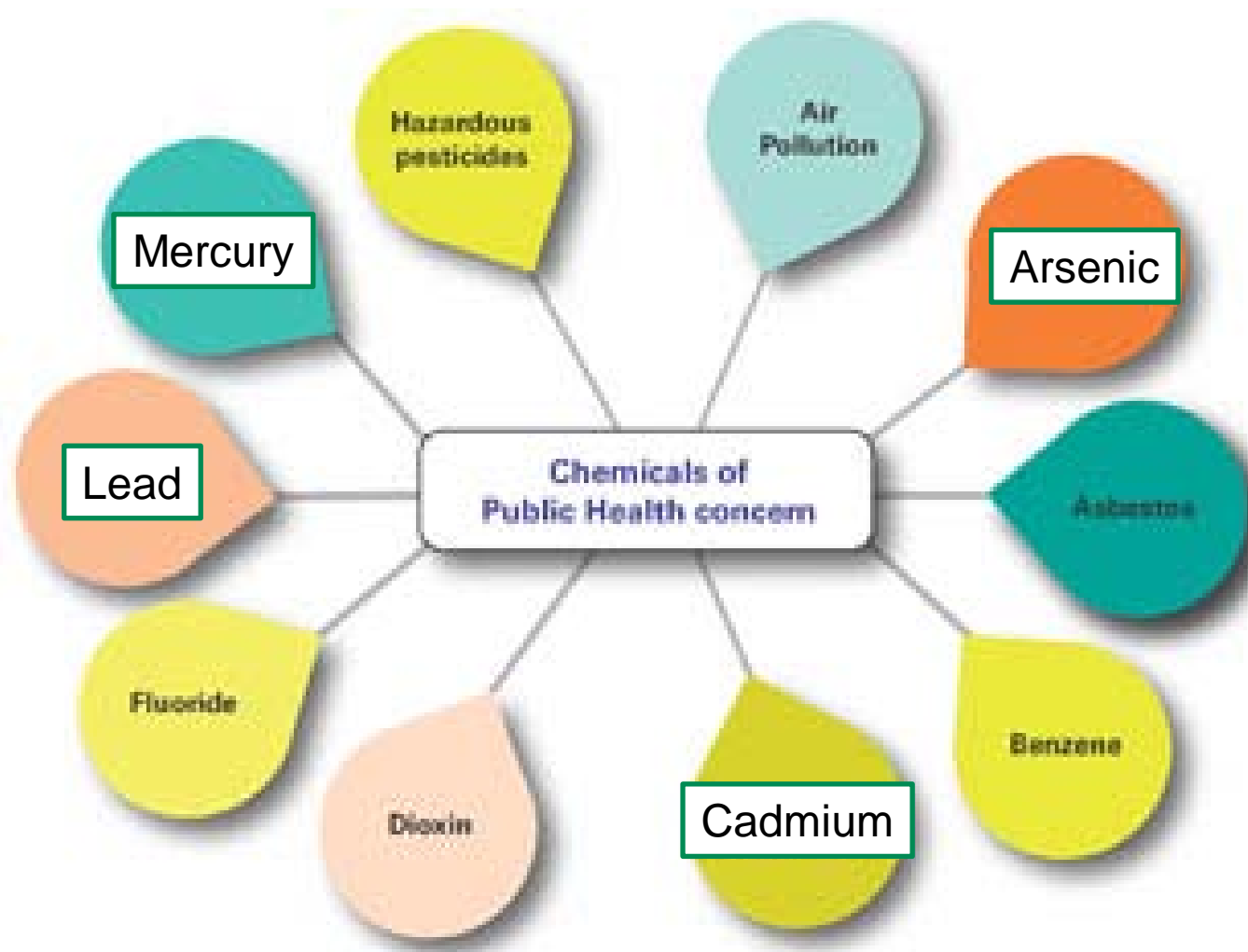
National Food Institute (DTU Food)
Technical University of Denmark



Agenda



- Speciation in relation to food and feed safety – where are we now??
- Current legislation and standardisation issues
- Selected examples
 - Arsenic speciation analysis (importance of inorganic arsenic)
 - Organotin speciation analysis (food contact materials)
 - Selenium speciation analysis (food fraud!)
- Future developments and needs



Current situation in EU legislation:

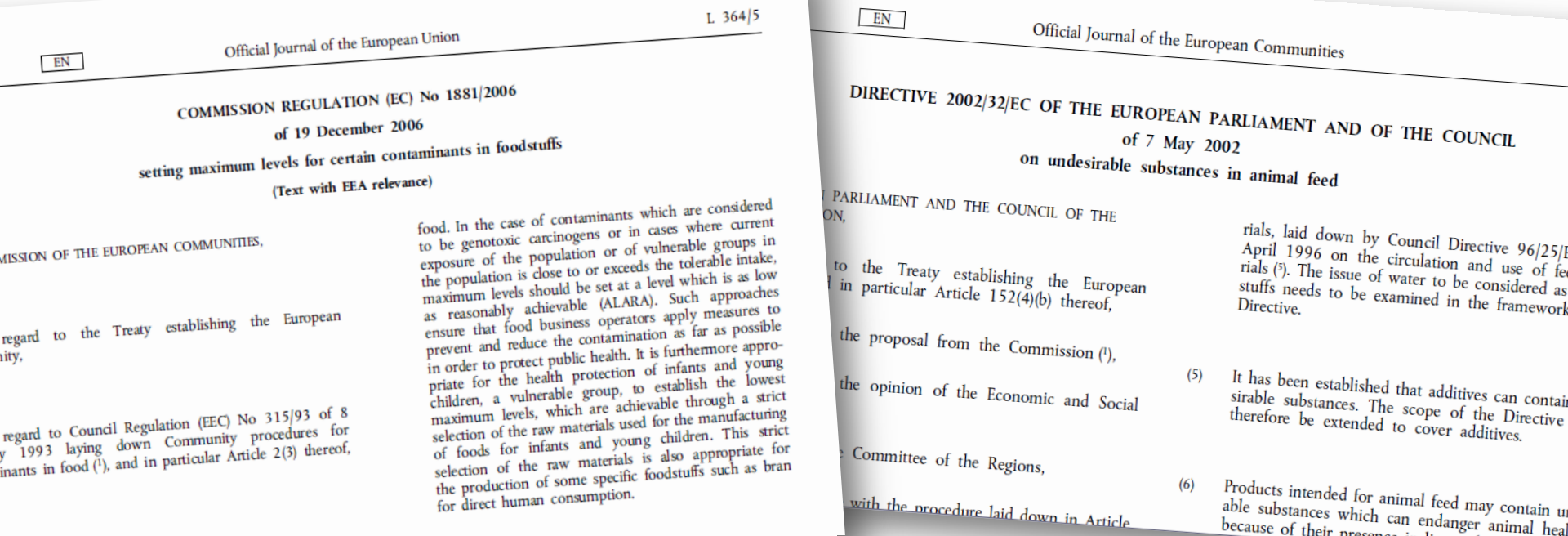
Foodstuffs

MLs for Pb, Cd, Hg and Sn
EU directive 2006/1881/EC

Animal feedingstuffs

MLs for As, Pb, Cd and Hg
EU directive 2002/32/EC

Only maximum levels for
total concentration of the
metals



Speciation and regulation - some historical viewpoints



1998

SPECTROCHIMICA
ACTA
PART B

Spectrochimica Acta Part B 53 (1998) 169–175

Speciation analysis: where is it going? An attempt at a forecast¹

Bernhard Welz

Department of Applied Research, Bodenseewerk Perkin-Elmer GmbH, D-4

Fresenius J Anal Chem (1999) 363:431–434

© Springer-Verlag 1999

CONFERENCE CONTRIBUTION

1999

Torsten Berg · Erik H. Larsen

**Speciation and legislation –
Where are we today and what do we need for tomorrow?**

Handbook of Elemental Speciation II

Species in the Environment,
Food, Medicine and
Occupational Health

Speciation and the Emerging Legislation

Nicole Proust

Editor-in-Chief *THALES Research and Technology France, Orsay, France*

Rita Cornelis

Wolfgang Buscher

Associate Editors *University of Münster, Münster, Germany*

Joe Caruso

Helen Crews Michael Sperling

Klaus Heumann *University of Münster, Münster, Germany*

Wiley 2005

SECTION: FORUM

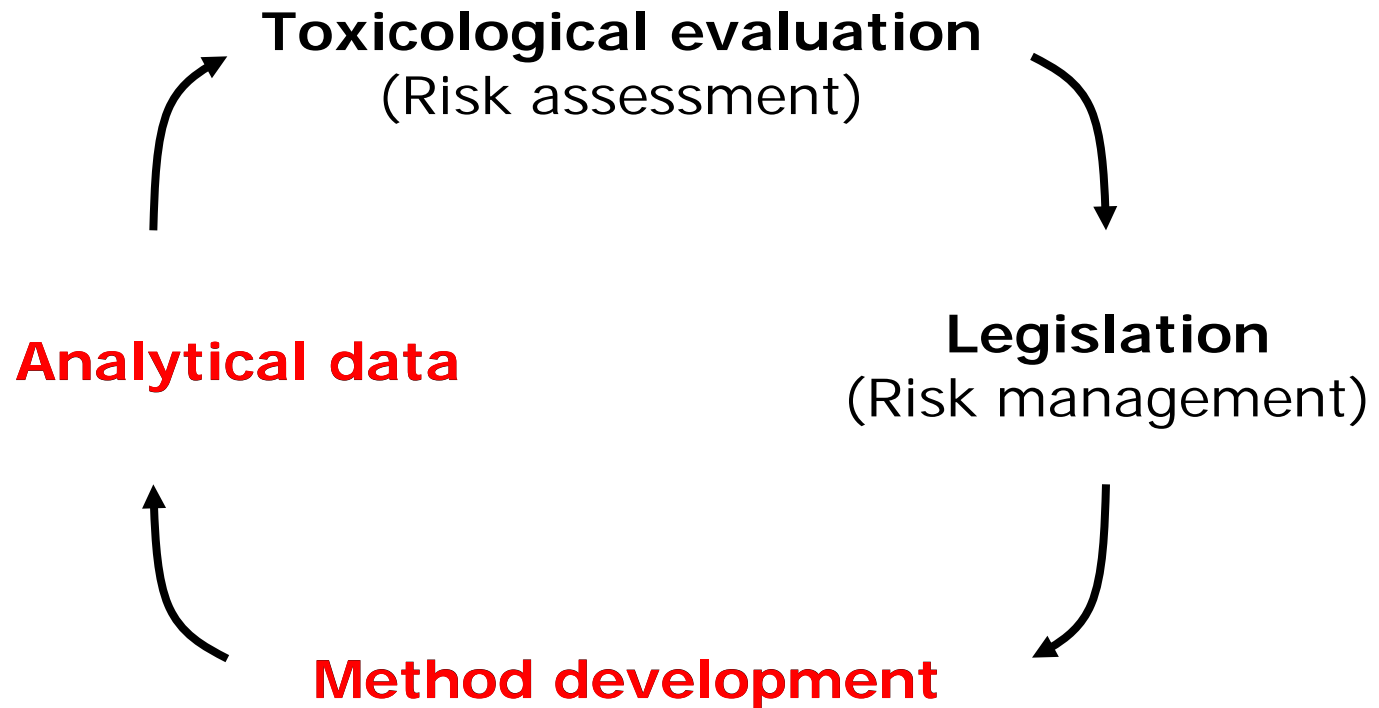
www.rsc.org/analyst | The Analyst

**Toxic metal species and food regulations—
making a healthy choice**

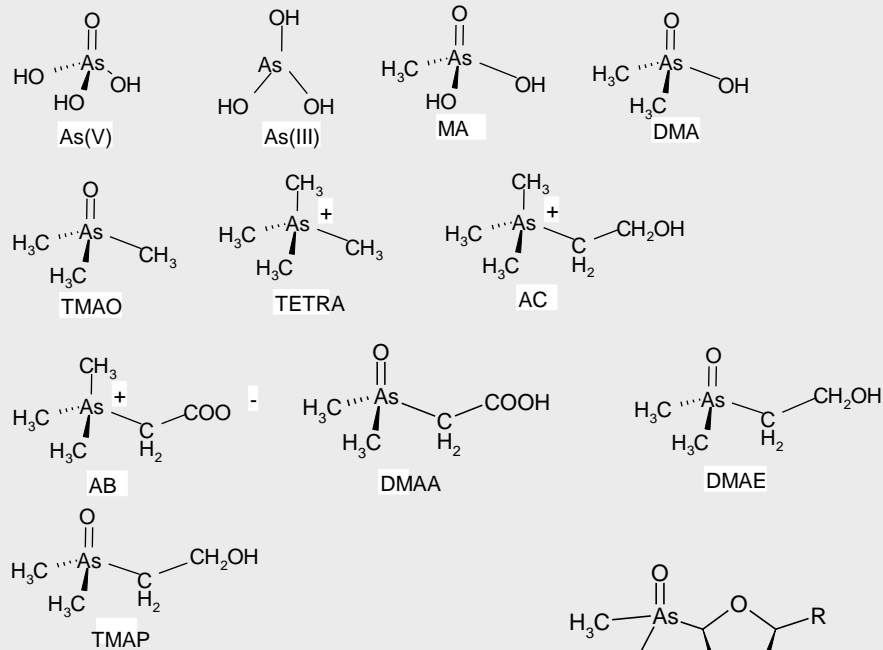
Kevin A. Francesconi

2007

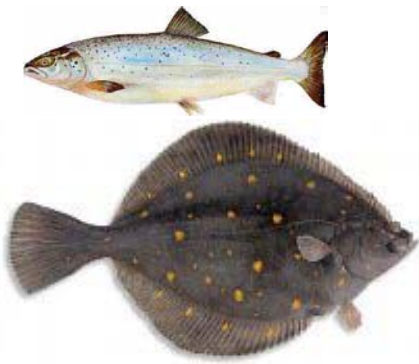
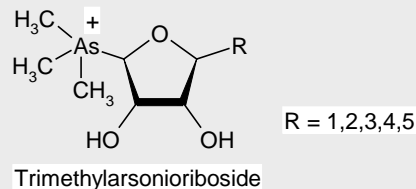
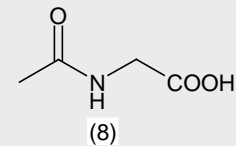
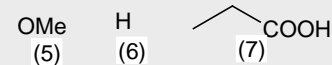
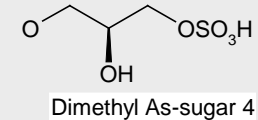
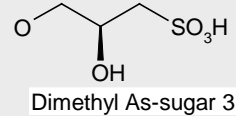
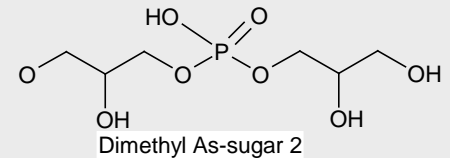
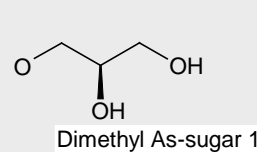
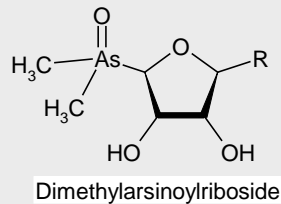
Vicious circle of progress



Arsenic compounds in the marine environment



More than **50** different arsenic species have been found in the marine environment – incl. lipid-soluble As compounds (*arsenolipids*).



Example – arsenic speciation

Important for correct risk assessment



1 kg rice => 50-300 μg As



1 kg fish => 3000-10000 μg As

There is most focus on rice from a food safety point of view – why???



The chemical form of arsenic is important
=> Arsenic speciation

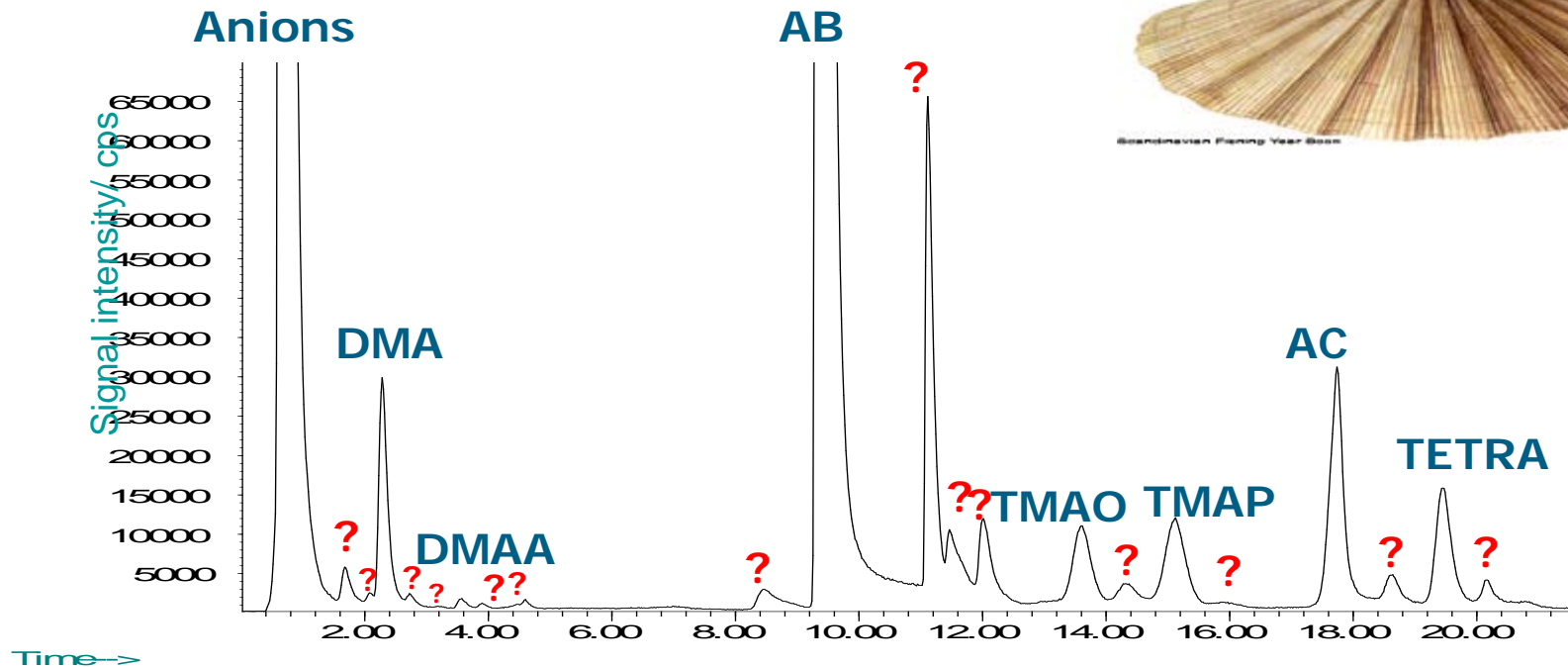
**Focus
on
Food
Safety**

Speciation analysis of arsenic of scallop kidney

Cation-exchange with gradient elution – extraction with aqueous methanol

Column: Chrompack Ionospher 5C; Mobile phase: Pyridine; pH = 2.7

- seven compounds identified by coelution with available standards
- **16** non-identified peaks



Food safety and arsenic – toxicity

Focus on inorganic arsenic; As(III) and As(V)

Long term exposure => skin diseases

- Keratosis, gangrene, melatosis
- Skin cancer
- ... and also
- lung, kidney, liver, bladder cancers



Cancer slope factor: $1.5 \text{ (mg kg}^{-1} \text{ day}^{-1})^{-1}$ (for inorganic As) (US EPA 2005)

~~WHO (1988) PTWI for inorganic arsenic: $15 \text{ } \mu\text{g/kg bw/week}$~~
(Provisional Tolerable Weekly Intake)

~~For a 70 kg person => $150 \text{ } \mu\text{g / day}$~~

No longer appropriate

EFSA (2009) and JECFA (2010) opinions on arsenic in food

- Old PTWI value (WHO, 1988) was withdrawn
- **NEW!** $BMDL_{1.0} = 0.3 - 8 \mu\text{g/kg bw per day}$ for inorganic arsenic
- => EU dietary exposures within this range
- => Risk to some consumers cannot be excluded



- **NEW!** $BMDL_{0.5} = \underline{3 \mu\text{g/kg bw per day}}$ for inorganic arsenic
=> *0.5% increased incidence of lung cancer for 12 y exposure*



- “...there is a need to produce speciation data for different food commodities to support dietary exposure assessment...”
- “...more accurate information on the inorganic arsenic content of foods is needed to improve assessments of dietary exposures to inorganic arsenic”
- “...need for validated methods for selective determination of inorganic arsenic in food matrices”

Sample identification	Inorganic arsenic	Total arsenic
Salmon (<i>Salmo salar</i>)	< 0.0006	1.9 0.2
Cod (<i>Gadus morhua</i>)	< 0.0006	17 2
Cod (<i>Gadus morhua</i>)	< 0.0006	15 2
Wolffish (<i>Anarhichas lupus</i>)	< 0.0006	4.1 0.5
Wolffish (<i>Anarhichas lupus</i>)	< 0.0006	31 4
Anglerfish (<i>Lophius piscatorius</i>)	< 0.0006	15 2
Anglerfish (<i>Lophius piscatorius</i>)	< 0.0006	44 6
Atlantic halibut (<i>Hippoglossus hippoglossus</i>)	< 0.0006	12 1
Mackerel (<i>Scomber scombrus</i>)	< 0.0006	1.7 0.2
Mackerel (<i>Scomber scombrus</i>)	< 0.0006	2.8 0.4
Herring (<i>Clupea harengus</i>)	< 0.0006	1.5 0.2
Herring (<i>Clupea harengus</i>)	< 0.0006	1.7 0.2
Herring (<i>Clupea harengus</i>)	< 0.0006	1.7 0.2
Tuna fish (<i>Thunnus alalunga</i>)	0.008 0.001	0.9 0.1
Lobster, tail meat (<i>Homarus gammarus</i>)	< 0.0006	14 2
Lobster, head and thorax meat (<i>Homarus gammarus</i>)	0.017 0.005	22 3
Crab, white meat (<i>Cancer pagurus</i>)	0.016 0.002	12 1
Crab, head and thorax meat (<i>Cancer pagurus</i>)	0.016 0.009	26 3
King crab, white meat (<i>Paralithodes camtschaticus</i>)	0.005 0.001	26 3
Norway lobster (<i>Nephrops norvegicus</i>)	0.020 0.003	21 5
Shrimp (<i>Pandalus borealis</i>)	< 0.0006	3.8 0.5
Shrimp (<i>Pandalus borealis</i>)	< 0.0006	60 8
Shrimp (<i>Pandalus borealis</i>)	< 0.0006	67 8
Horse mussel (<i>Modiolus modiolus</i>)	0.0012 0.002	3.4 0.4
Scallop muscle (<i>Pecten maximus</i>)	0.008 0.001	3.1 0.3
Oyster (<i>Ostrea edulis</i>)	0.014 0.002	1.8 0.2
Mink whale (<i>Balaenoptera Acutorostrata</i>)	< 0.0006	0.61 0.08
Harp seal (<i>Pagophilus groenlandicus</i>)		0.9 0.1
Hooded seal (<i>Cystophora cristata</i>)		0.22 0.03

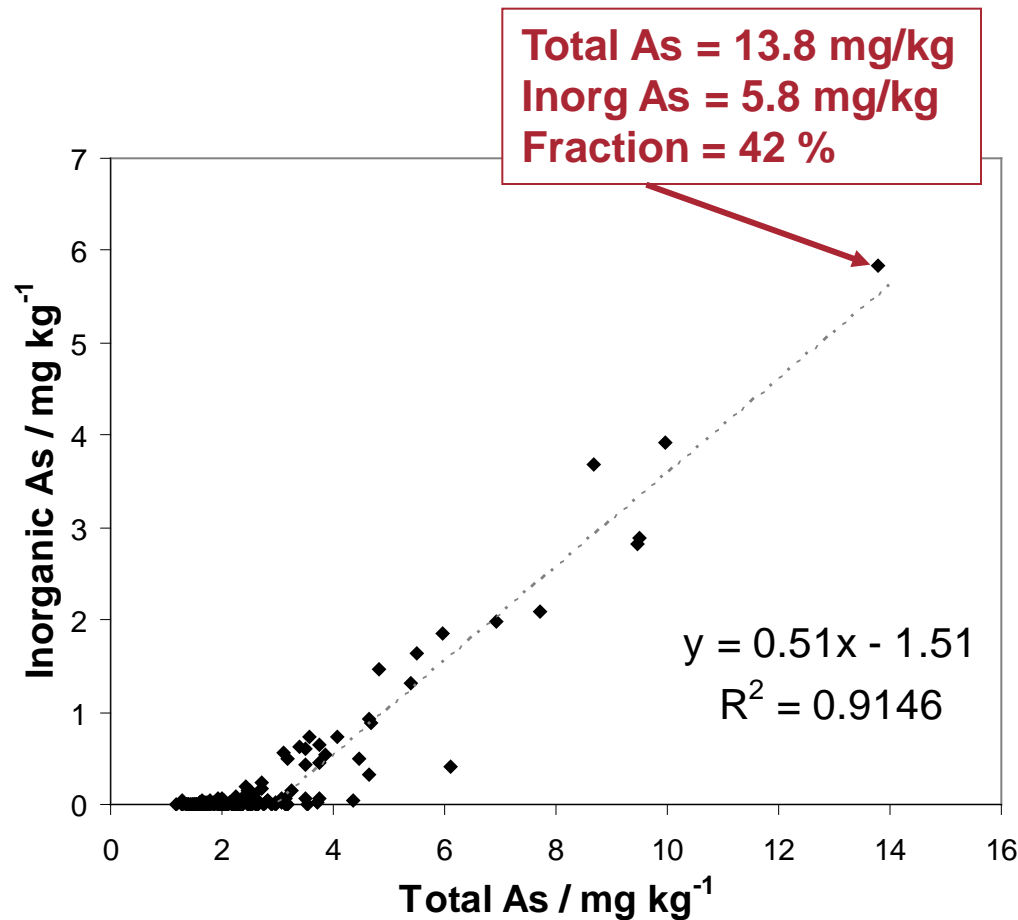
Fish muscle

Crustaceans
& bivalves

Marine mammals

For all samples inorganic arsenic constitutes less than 1% of total arsenic

...but unusual high contents in some samples...



Data from 175 blue mussel (*Mytilus edulis*) samples collected along the Norwegian Coastline.



Arsenic in rice – an emerging health issue?

Environmental Pollution 152 (2008) 746–749

Rapid communication

Inorganic arsenic levels in baby rice are of concern

Andrew A. Meharg^{a,*}, Guoxin Sun^b, Paul N. Williams^{a,b}, Eureka Adomako^a,
Claire Deacon^a, Yong-Guan Zhu^b, Joerg Feldmann^c, Andrea Raab^c

^a School of Biological Sciences, University of Aberdeen, Cruickshank Building, St. Machar Drive, Aberdeen AB24 3UU, UK

^b Research Centre for Eco-Environmental Sciences, Chinese Academy of Sciences, Beijing 100085, China

Median consumption of organic arsenic levels for UK babies from baby rice is above threshold considered safe.

- 17 samples from supermarkets in Aberdeen
- Total arsenic levels: 0.12 – 0.47 mg/kg
- Inorganic arsenic: 0.06 – 0.16 mg/kg (33 – 69 % of tAs)
- 35% above Chinese max level of 0.15 mg/kg iAs
- No regulation on As in food in EU (yet!)

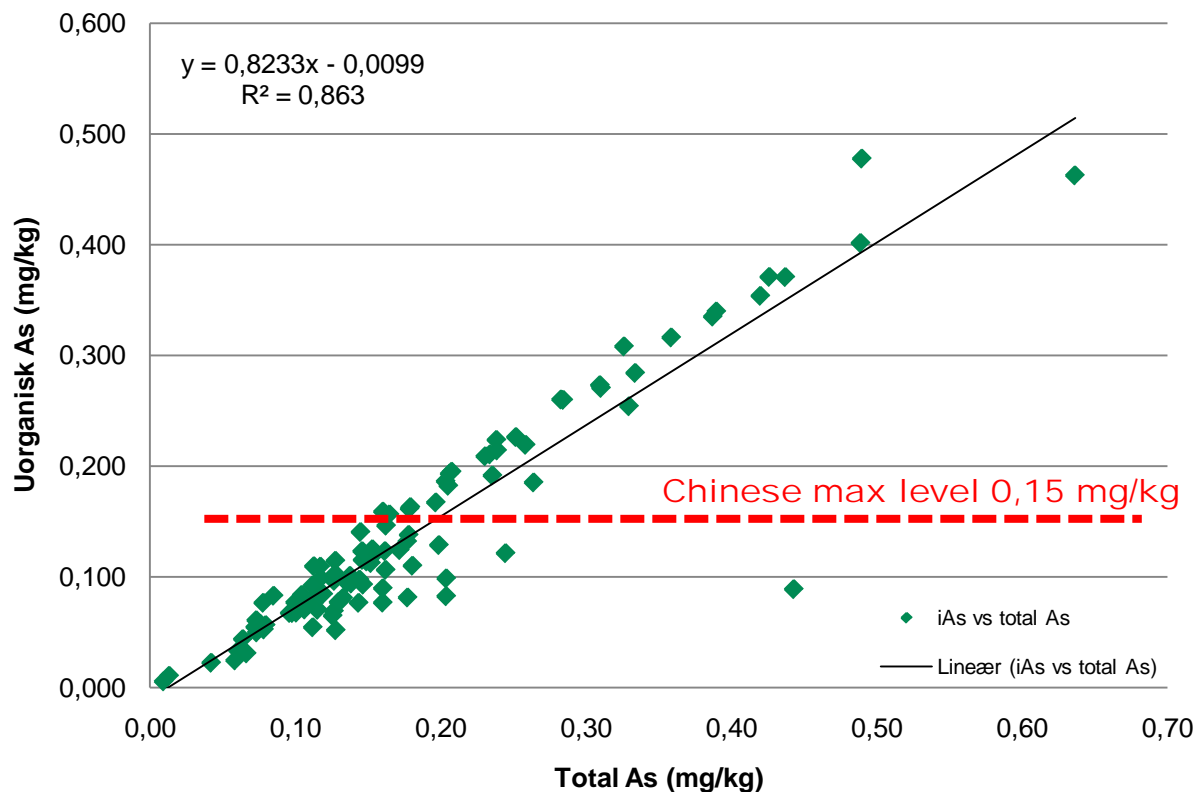


Comment on cereals Cubadda



Arsenic in rice products

iAs vs total As



105 prøver i alt

- Hvide ris (white rice)
- Brune ris (brown rice)
- Røde ris (red rice)
- Sorte ris (black rice)
- Ris kiks (rice crackers)

33 prøver > 0,15 mg/kg

- 2 parboiled (20%)
- 4 brune (50%)
- 4 røde (50%)
- 5 sorte (71%)
- 1 Basmati (10%)
- 1 Grød (9%)
- 1 vilde (20%)
- 15 ris kiks (100%)

Rice cracker mean: 0,31 mg/kg – intake 50 g/dag => 15 µg iAs (~1 µg/kg)
> EFSA BMDL₀₁ på 0,3-8 µg/kg bw/dag

Inorganic arsenic in chinese food supplements

Name of Food supplement	Total Arsenic ($\mu\text{g/g}$)	Inorganic arsenic ($\mu\text{g/g}$)
Xiao Yao Wan	0.82	0.85
Bu Zhong Yi Qi Wan	0.62	0.50
Da Bu Yin Wan	0.59	0.55
Six Flavor teapills	0.72	N.D.
Golden Book Teapills	0.58	0.57
Xiang Sha Liu Jun Zi Wan	0.94	0.80
Gan Mao Ling	1.24	1.01
Chuan Xin Lian	5.00	3.17
Bi Yan Pian	0.70	0.58
Arouse power	1.12	1.02
Bio Chlorella	0.62	0.21
Unik Spirulina Kapsler	2.59	0.13
Chlorella	0.58	0.03
Ez-Biloba	0.63	0.67
Qvinde Dong Quai	0.68	0.48



Gan Mao Ling
 Rec dose: 18 pills per day
 \Rightarrow iAs $\sim 13 \mu\text{g/day}$
 $\Rightarrow 0.22 \mu\text{g/kg bw/day}$ (60 kg)
 Close to EFSA BMDL !!



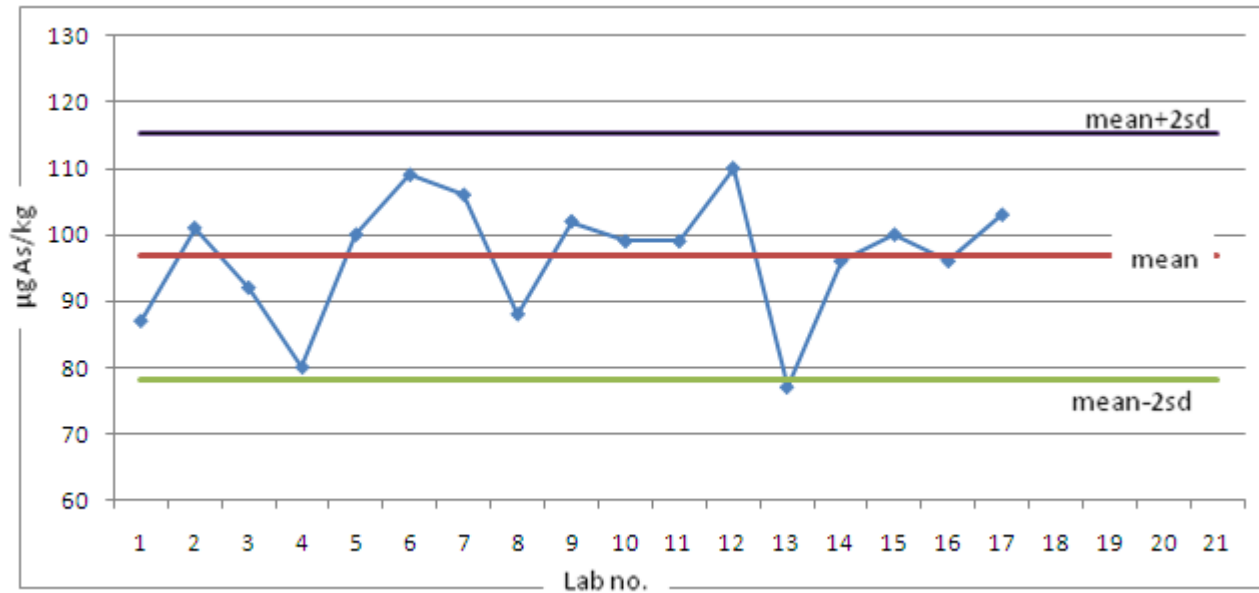
Recent meeting: the Expert Committee “Industrial and Environmental Contaminants”

“Based on the data processed so far, a maximum level of 0.2 mg/kg inorganic arsenic for rice as a category seems achievable”

“MSs are asked to reflect on the need for a maximum level for cereals other than rice”

“MSs are encouraged to concentrate on levels for inorganic arsenic in rice (collecting information on country of origin and rice variety), food supplements (algae) and infant food (rice based)”

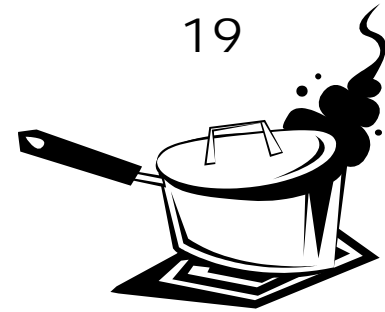
Inorganic As in SRM NIST1586a



Extraction solution	Detection	iAs (µg/kg)	Reference
1 2M TFA	LC-ICPMS	87 +/- 9	Ackermann (2005)
2 Enzymatic, pepsin and pancreatin	LC-ICPMS	101 +/- 7	Ackermann (2005)
3 2M TFA	LC-ICPMS	92 +/- 2	Heitkemper (2001)
4 2M TFA	LC-ICPMS	80 +/- 16	Williams (2005)
5 2M TFA	LC-ICPMS	100 +/- 10	Williams (2006)
6 MeOH:H2O	LC-ICPMS	109 +/- 3	D'Amato (2004)
7 Enzymatic, alfa-amylase	LC-ICPMS	106 +/- 7	Kohlmeyer (2003)
8 Enzymatic, protease and alfa-amylase	LC-ICPMS	88 +/- 6	Sanz (2005)
9 1M H3PO4	HG-AFS	102 +/- 2	Matosreyes (2007)
10 1% HNO3	LC-ICPMS	99 +/- 4	Raab (2009)
11 1% HNO3	LC-ICPMS	99 +/- -	Sun (2008)
12 1% HNO3	LC-ICPMS	110 +/- 10	Sun (2009)
13 0,5 M TFA	LC-ICPMS	77 +/- -	Heitkemper (2009)
14 enzymatic, proteas and alfa-amylase	LC-ICPMS	96 +/- 9	Mar (2009)
15 2M TFA	LC-ICPMS	100 +/- 12	Meharg (2008)
16 water	LC-ICPMS	96 +/- 3	Narukawa (2008)
17 0,07M HCL and 10%H2O2	LC-ICPMS	103 +/- 15	DTU Food (2009)

Good agreement
between
labs and methods

+ lecture by M. de la Calle



Processing

- Processing or storage may alter the arsenic species pattern
- No transformation of organoarsenic compounds to inorganic arsenic by normal cooking procedures (**IMPORTANT!**)
- Loss/uptake to/from boiling water possible
- Blue mussels => loss of AB after storage by freezing



Commission directive 2009/114/EC (amendment)

Undesirable substances	Products intended for animal feed	Maximum content in mg/kg (ppm) relative to a feedingstuff with a moisture content of 12 %
(1)	(2)	(3)
1. Arsenic (*) (**)	Feed materials with the exception of:	2
	— meal made from grass, from dried lucerne and from dried clover, and dried sugar beet pulp and dried molasses sugar beet pulp,	4
	— palm kernel expeller,	4 (***)
	— phosphates and calcareous marine algae,	10
	— calcium carbonate,	15
	— magnesium oxide,	20
	— feedingstuffs obtained from the processing of fish or other marine animals, including fish,	25 (***)
	— seaweed meal and feed materials derived from seaweed,	40 (***)
	Iron particles used as tracer.	50
	Additives belonging to the functional group of compounds of trace elements except:	30



Only max levels for total arsenic

FOOTNOTE

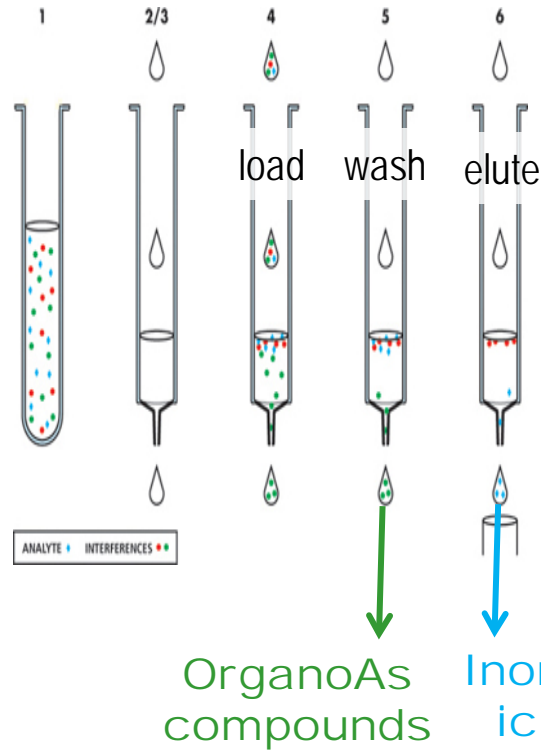
(***) Upon request of the competent authorities, the responsible operator must perform an analysis to demonstrate that the content of inorganic arsenic is lower than 2 ppm in the following products: (a) complete feedingstuffs for fish and complete feedingstuffs for fur animals; (b) complementary feedingstuffs with the exception of mineral feedingstuffs.

Inorganic arsenic lower than 2 ppm

Complete feedingstuffs with the exception of:	2
— complete feedingstuffs for fish and complete feedingstuffs for fur animals,	10 (***)
Complementary feedingstuffs with the exception of:	4
— mineral feedingstuffs,	12

Speciation analysis is required !!

SPE-HG-AAS – a speciation alternative...



Sequential elution for selective off-line separation of inorg As from organo As species by SPE

Abundance

75000
70000
65000
60000
55000
50000
45000
40000
35000
30000

Sample load

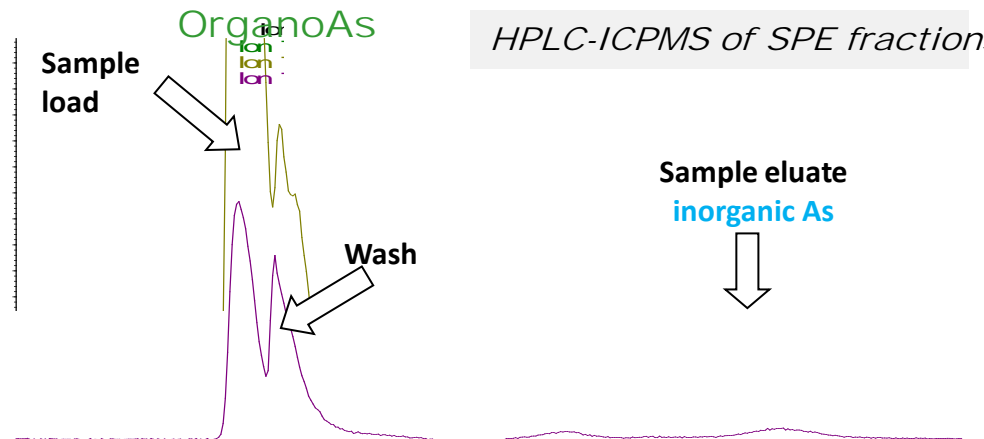
OrganoAs

ion
ion
ion

Wash

HPLC-ICPMS of SPE fractions

Sample eluate
inorganic As



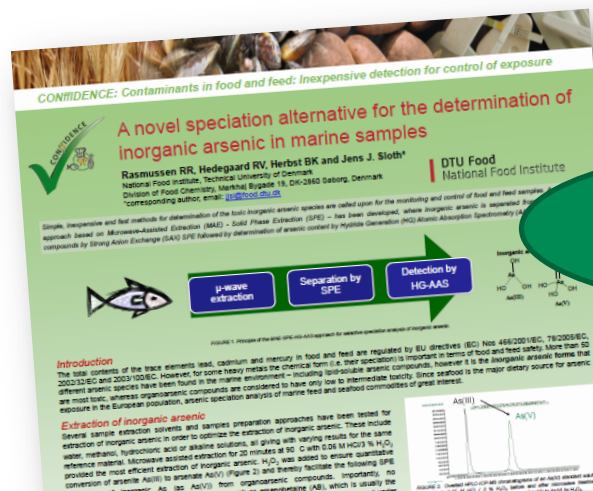
Performance characteristics from in-house validation

μ-wave
extraction

Separation by
SPE

Detection by
HG-AAS

Parameter	Result
Analysis time	2 x 7 h for 24 samples
LoD (mg/kg)	0.08
LoQ (mg/kg)	0.16
Repeatability (%RSD)	3 - 7
Accuracy (%)	90 - 104



Further details on
poster and handouts

Samples for collaborative trial

Sample no	Sample type	Spiked / unspiked	Mean concentration (mg Kg ⁻¹)
IMEP32-1	Complete fish feedingstuff	Unspiked	0.07
IMEP32-2	Complete fish feedingstuff	Spiked	0.71
IMEP32-3	Fish meal	Unspiked	0.19
IMEP32-4	Fish meal	Spiked	1.06
IMEP32-5	Fish fillet	Spiked	2.64
IMEP32-7	Fish meal	Spiked	0.43
IMEP32 Control sample	CRM TORT-2 Lobster Hepatopancreas	Unspiked	0.54

Control sample

Concentration range: 0.07 – 2.64 mg Kg⁻¹



National Research
Council Canada

Conseil national
de recherches Canada

TORT-2

**Lobster Hepatopancreas Reference Material
for Trace Metals**

Trace Metals
(milligrams/kilogram)

Arsenic (g,h,m) 21.6 ± 1.8

- Total As
- Not certified for iAs

Results - overview

Matrix	Units	IMEP 32-1	IMEP 32-2	IMEP 32-3	IMEP 32-4	IMEP 32-5	IMEP 32-7	IMEP 32 Control Sample
N° of participating laboratories		10	10	10	10	10	10	10
Remaining data after outlier elimination		29	35	28	36	36	30	34
N° of remaining laboratories		9	10	9	10	10	9	10
Outliers	%	12.1	2.8	12.5	0.0	0.0	11.8	0.0
Overall mean $X_{obs} \pm u_{obs}$	mg Kg ⁻¹	0.071 ± 0.041	0.713 ± 0.117	0.189 ± 0.060	1.062 ± 0.140	2.643 ± 0.506	0.432 ± 0.066	0.544 ± 0.162
S_r	mg Kg ⁻¹	0.016	0.054	0.014	0.105	0.277	0.023	0.095
RSD_r	%	22.8	7.6	7.5	9.9	10.8	5.4	17.5
r_L	mg Kg ⁻¹	0.046	0.153	0.040	0.294	0.776	0.065	0.266
S_R	mg Kg ⁻¹	0.041	0.117	0.060	0.140	0.506	0.066	0.162
RSD_R	%	57.6	16.4	31.9	13.2	19.1	15.3	29.7
R_L	mg Kg ⁻¹	0.115	0.327	0.169	0.391	1.416	0.185	0.453
σ_H	mg Kg ⁻¹	0.017	0.120	0.039	0.168	0.365	0.078	0.095
HorRat		2.4	1.0	1.6	0.8	1.4	0.8	1.7



HorRat > 2
Low concentration!!

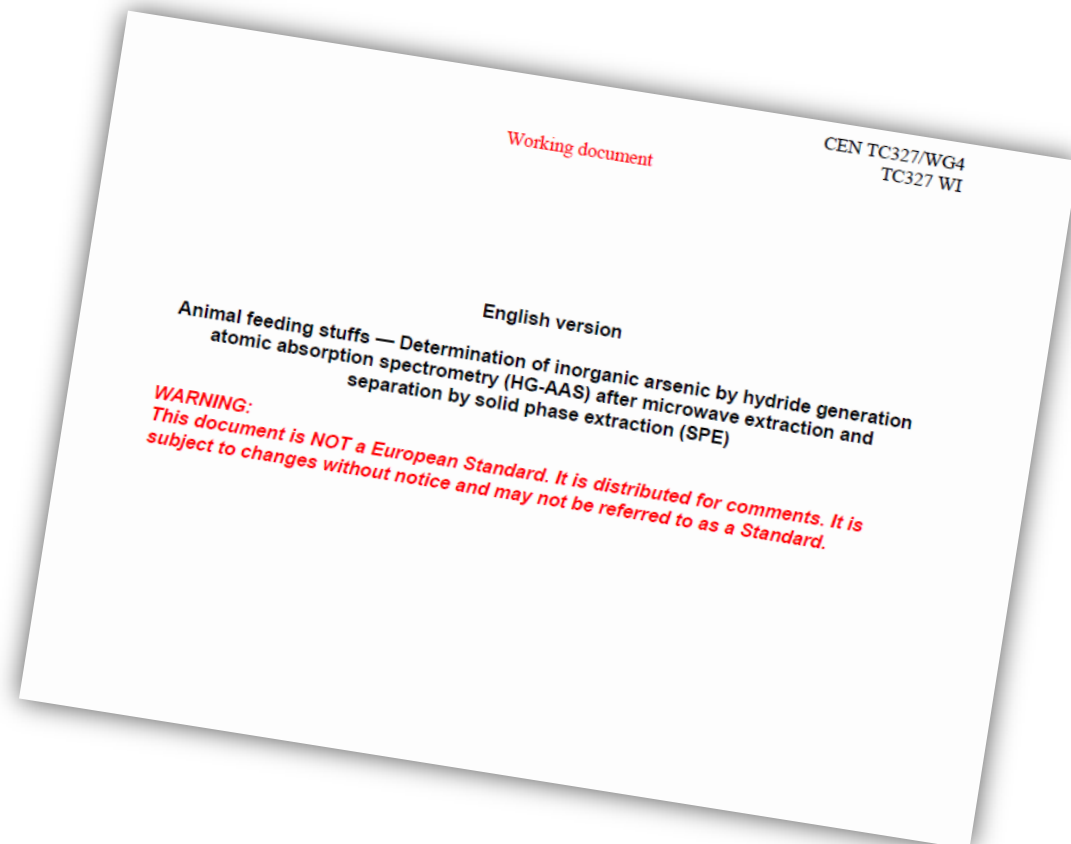
All HorRat values < 2

Conclusions - I

- A method for determination of iAs in feed of marine origin based on SPE-HG-AAS has been developed
- homogeneous and stable test samples were prepared
- a collaborative trial was conducted in Oct-Nov 2010
- Ten laboratories were evaluated as compliant
- Max 1 outlier lab was identified per sample
- HorRat values < 2 (0.8 – 1.7) for samples in the concentration range 0.19 – 2.6 mg Kg⁻¹
- HorRat value > 2 (2.4) for S01 at concentration 0.07 mg Kg⁻¹
- -Accuracy from control sample, mean = 0.544 ± 0.162 mg Kg⁻¹
-Assigned value from HPLC-ICPMS determinations = 0.599 ± 0.07 mg Kg⁻¹
- $0.544/0.599 = 91\%$ (no significant difference)

Conclusions - II

- EU directive "guideline" maximum level is 2 ppm (EU directive 2009/141/EC on animal feed)
- Method working range tested in ILC: 0.1 – 2.6 mg Kg⁻¹
- HorRat values <2 in the working range tested
- The method is fit for purpose



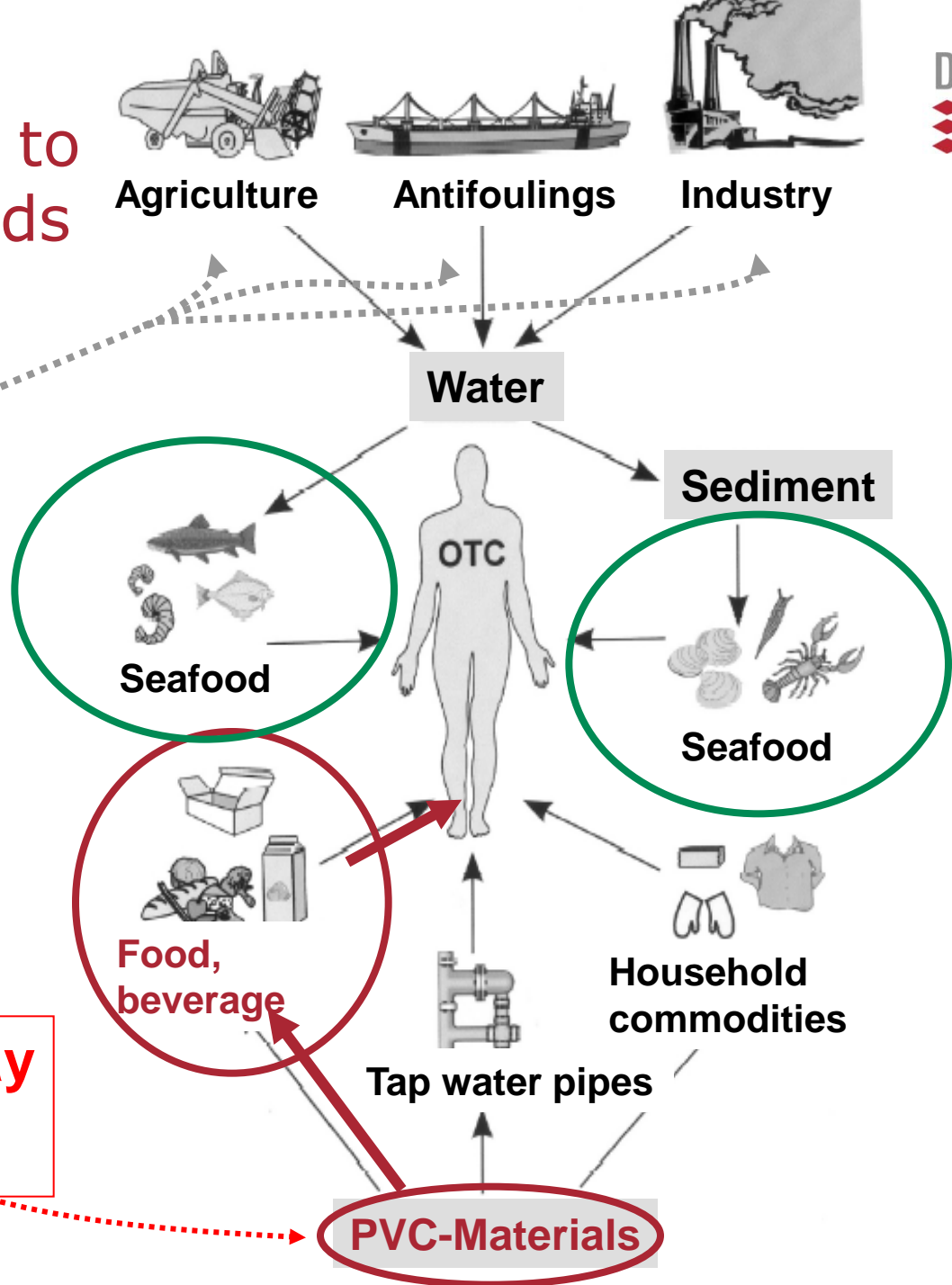
Routes of exposure to organotin compounds

Used in

- Agriculture
- Antifoulings
- Industry

- PVC-Materials

TDI: 0.25 $\mu\text{g}/\text{kg bw}/\text{day}$
 \sum TBT, DBT, TPhT and DOT



Legislation on OTCs in Food Contact Materials



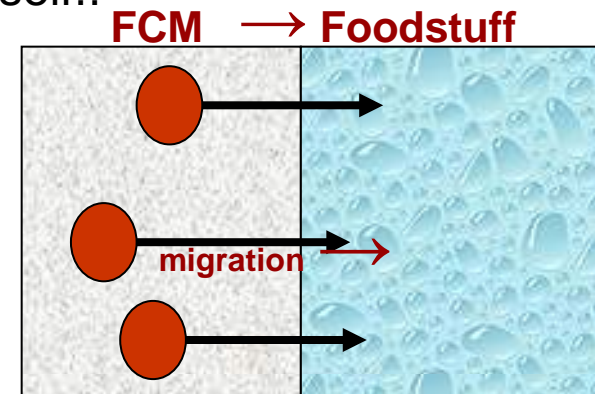
Compounds	Maximum level ($\mu\text{g Sn/kg}$ foodstuff)
\sum DBT, TBT, TPhT and DOT	40 (6)
\sum MMT, DMT	180
MOT	1200
MDDT	12000 (50)
DDDT	24000 (50)

Ref: EFSA (2005); proposed EFSA values in parenthesis

Assumptions:

- 1 kg food per 6 dm²
- 100 mL in contact with 0.6 dm²

- Max levels on organotin migrating from the packaging material
- Testing by the use of food simulators (water, acid, oil, alcohol etc)
- BUT no maximum levels on organotins in the foodstuff itself!!

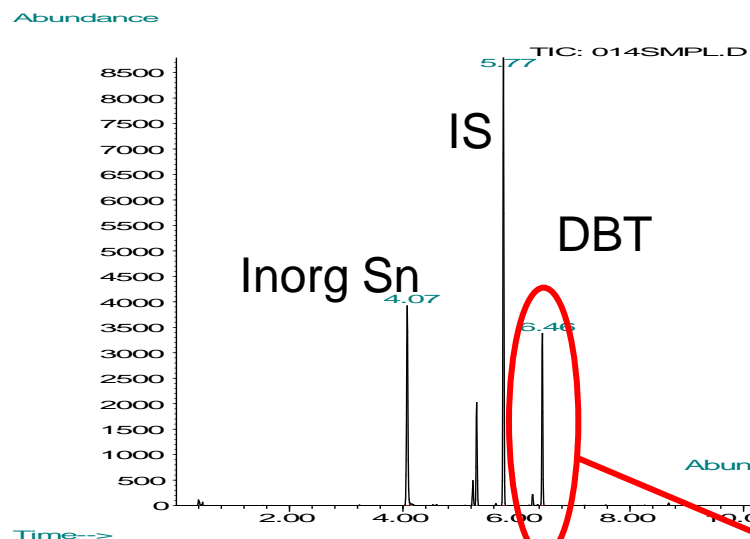


Organotin migration from Food Contact Materials II

Small scale survey on 33 FCMs

Baking paper, PVC cling films, silicone baking forms, lids with PVC gaskets

PUR-agglomerated cork wine stoppers



- PVC lid
- 3% acetic acid

Overlaid standard and sample

➤ DBT concentration: 9.9 µg/kg

TIC: 005SMPL.D
TIC: 014SMPL.D

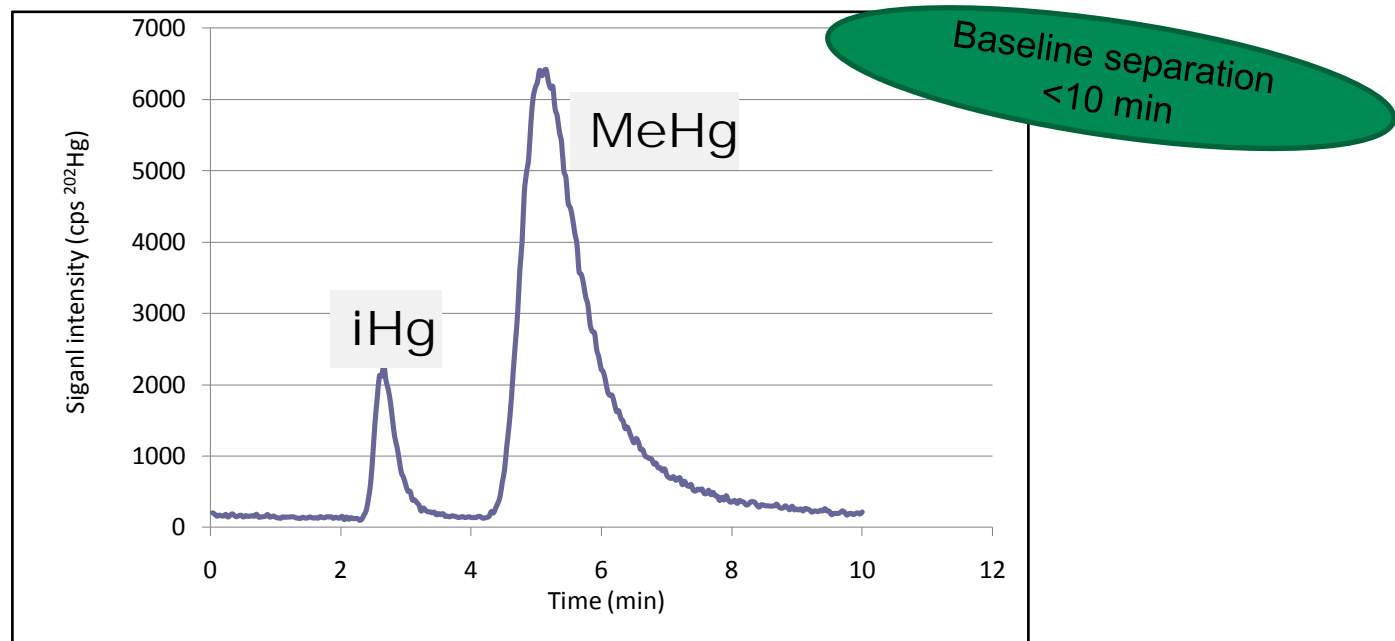
DBT

> EFSA guideline value of 6 µg/kg

Output of DK survey:

- 33 samples
- 11 contained OT (mainly DBT)
- 3 exceeded EFSA guideline limit

Speciation analysis of Mercury by HPLC-ICPMS



HPLC-ICPMS chromatogram of DORM-3 (Dogfish muscle)

CRM	Certified (mg/kg)	Result (mg/kg)
DORM-2 (dogfish muscle)	4.47 +/- 0.32	4.21
DORM-3 (dogfish muscle)	0.355 +/- 0.056	0.35
TORT-2 (Lobster hepatopancreas)	0.152 +/- 0.013	0.16

Relevance for regulation ?? But Codex opinion

Selenium in commercial food supplements

Organic bound Se?

Se yeast?

Selenite?

Selenate?

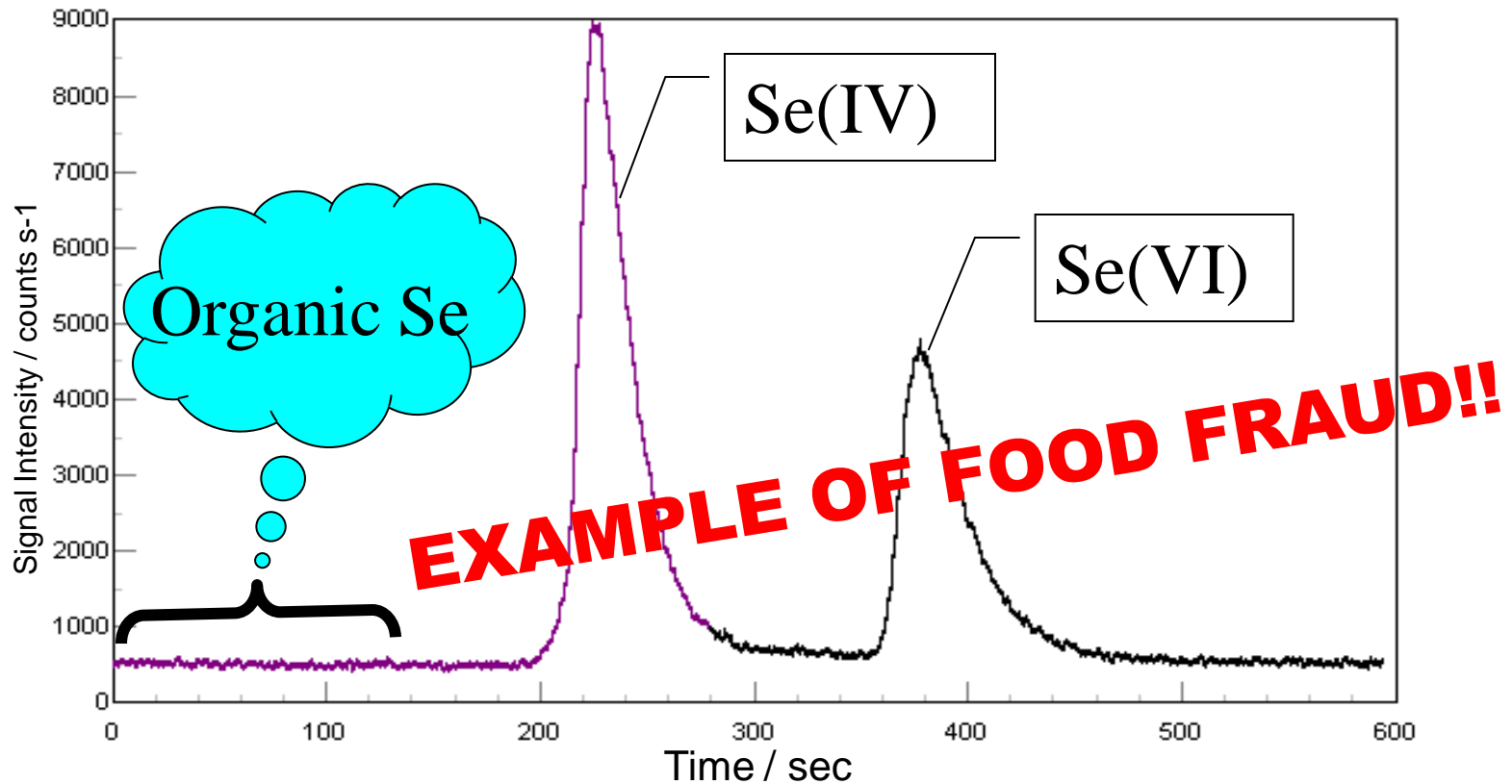
Selenomethionine?

Amino chelated Se?



Se speciation by HPLC-ICPMS

Supplement declared as organic bound Selenium (125 µg/tablet)



Larsen, unpublished data.

Identification number of the additive	Name of the holder of authorisation	Additive	Composition, chemical formula, description, analytical method	Species or category of animal	Maximum age	Minimum content	Maximum content	Other provisions
						Maximum content of element (Se) in mg/kg of complete feedstuff with a moisture content of 12 %		
Category of nutritional additives. Functional group: compounds of trace elements								
3b8.12	—	Selenomethionine Selenomethionine produced by <i>Saccharomyces cerevisiae</i> CNCM I-3399 (Selenized yeast inactivated)	Characterisation of the additive: Organic selenium mainly selenomethionine (63 %) content of 2 000-2 400 mg Se/kg (97-99 % of organic selenium) Characterisation of the active substance: Selenomethionine produced by <i>Saccharomyces cerevisiae</i> CNCM I-3399 (Selenized yeast inactivated) Analytical method ⁽¹⁾ : Zeeman graphite furnace atomic absorption spectrometry (AAS) or hydride AAS	All species	—		0,50 (total)	1. The additive shall be incorporated in to feed in form of a premixture. 2. For user safety: breathing protection, safety glasses and gloves should be worn during handling.

⁽¹⁾ Details of the analytical methods are available at the following address of the Community Reference Laboratory: www.imm.jrc.be/crl-feed-additives

Selenium in feed
Incurred content vs added

Organic bound minerals vs inorganic minerals
Feed additives and food supplements
EFSA opinions

Speciation summary

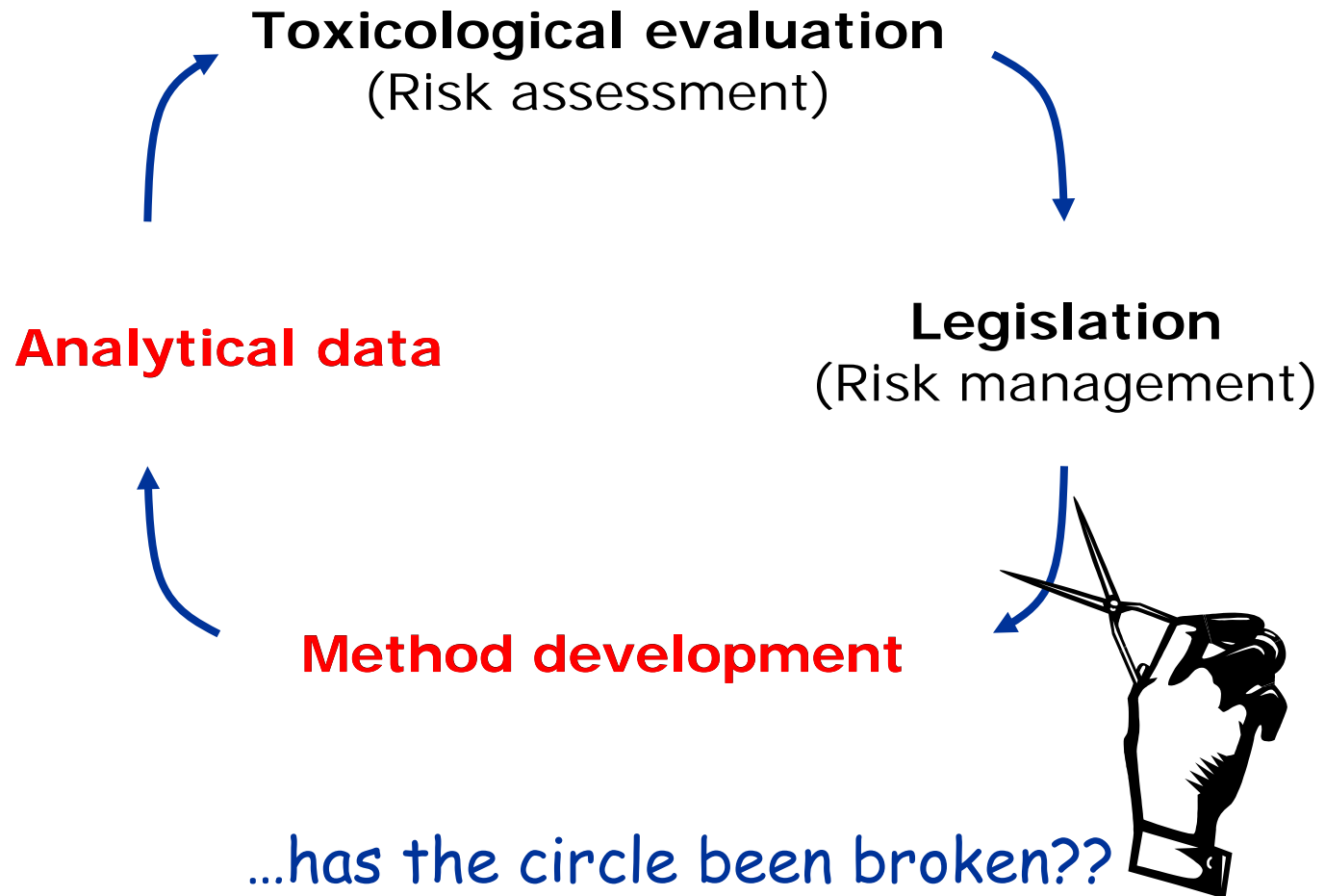


- ✓ speciation methods are more and more commonly used
- ✓ instrumentation is widely available
- ✓ legislation on species has started
- ✓ ...and more is expected in the future!



- ✓ standardised methods are not ready!
- ✓ ...but the need is known by authorities
- ✓ legislation is still behind!
- ✓ Lack of CRMs (e.g. for iAs)

...how to proceed?



Perspectives I

- Maximum levels in the legislation are needed
- Standardised methods for official control is needed
- On-site screening methods for fast answers
 - biosensor based => answer: result>ML?
 - can a batch of food/feed be released for trade or is further analysis required?
- Fraud cases in food supplements / feed additives
 - do you get what you pay for?

Perspectives II – trace elements in nanoform

AEROSIL® *Evonik*



silica nanoparticles as food additive for powdered food (e.g. soup, coffee creamer)

<http://www.aerosil.com/>



silver nanoparticles as food supplement
www.fairvital.com



nanoclay in PET beer bottles

www.honeywell.com

NanoLyse Project

"Nanoparticles in Food: Analytical methods for detection and characterisation"

Validated methods for the determination of inorganic ENP in food extracts, based on size separation, size determination and specific detection

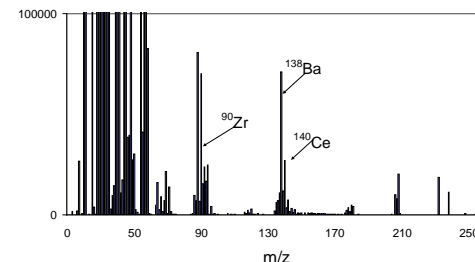
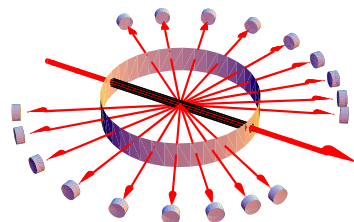
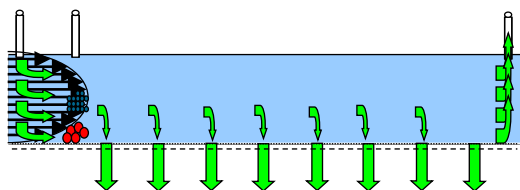
silver nanoparticles in lean meat



silica nanoparticles in tomato soup



Our platform: AF4-MALS/DLS-ICP-MS



asymmetric flow
field flow
fractionation
(AF4)

optical detection
multi angle (MALS)
and dynamic light
scattering (DLS), UV-
vis absorption and
fluorescence

inductively coupled
plasma mass
spectrometry
(ICP-MS)

particle separation
according to their
size (1nm – few μm)

particle detection
size determination

elemental detection
chemical identity
quantification

Schmidt, B.; Loeschner, K.; Hadrup, N.; Mortensen, A.; Sloth, J.J.; Koch, C.B. and Larsen, E.H.:
“Quantitative Characterization of Gold Nanoparticles by Field-Flow Fractionation Coupled Online with
Light Scattering Detection and Inductively Coupled Plasma Mass Spectrometry”, Analytical Chemistry,
vol. 83 (7), 2461-2468 (2011).

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